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Space Programs Summary No. 37-31, Volume V

for the period December 1, 1964 to January 31, 1965

Supporting Research and Advanced Development

(Title Unclassified)

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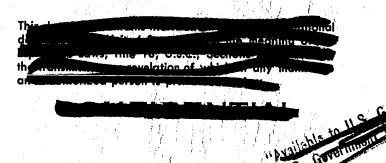
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PASADENA, CALIFORNIA

February 28, 1965



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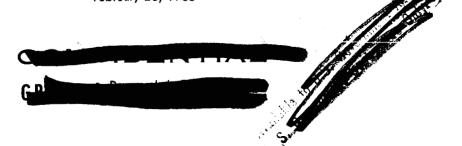
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Preface

The Space Programs Summary is a six-volume, bimonthly publication designed to report on JPL space exploration programs, and related supporting research and advanced development projects. The subtitles of all volumes of the Space Programs Summary are:

- Vol. I. The Lunar Program (Confidential)
- Vol. II. The Planetary-Interplanetary Program (Confidential)
- Vol. III. The Deep Space Network (Unclassified)
- Vol. IV. Supporting Research and Advanced Development (Unclassified)
- Vol. V. Supporting Research and Advanced Development (Confidential)
- Vol. VI. Space Exploration Programs and Space Sciences (Unclassified)

The Space Programs Summary, Volume VI consists of an unclassified digest of appropriate material from Volumes I, II, and III; an original presentation of the JPL (1) quality assurance and reliability efforts, and (2) environmental- and dynamic-testing facility-development activities; and a reprint of the space science instrumentation studies of Volumes I and II.

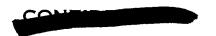
W. H. Pickering, Director Jet Propulsion Laboratory

Space Programs Summary No. 37-31, Volume V

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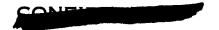
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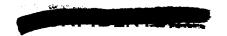


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PROPULSION DIVISION

I. Solid Propellant Engineering

A. Development of High-Energy Solid Propellant

F. A. Anderson

1. Introduction

The ultimate objective of this program is the complete characterization of hydrazine diperchlorate (HP₂) in order to (1) understand it chemically, (2) determine the conditions for its proper safe handling, (3) determine its compatibility with various organic materials, and (4) develop a high-performance solid propellant based on HP₂ as the oxidizer. To date moisture has been found to be the most critical single factor affecting the behavior of HP₂. This article discusses the methods of analyzing HP₂, the chemical and physical properties of different lots of HP₂, and some of the effects of moisture on the properties of HP₂.

2. Hydrazine Diperchlorate Analysis

Three different lots of HP₂ from two different sources are being studied. Lots NPP-1 and NPP-2 are both from the Naval Propellant Plant, Indian Head, Maryland. This

material was shipped in ½-lb containers in CCl₄. The third lot of HP₂, Lot TCC-1, was received from the Thiokol Chemical Corp. and was shipped as a dry powder. Several samples from Lot NPP-1 which were being stored in CCl₄ discolored and became dark gray, while other samples being stored in the same manner remained pure white. The results from an investigation into the probable causes for the discoloration strongly suggest that the discoloration is simply a surface effect caused by contamination by some silicone grease used in the apparatus during production. Table 1 shows the analysis of Lot NPP-1 along with the theoretical values for pure

Table 1. Analysis of hydrazine diperchlorate

V.	Theoretical	Lot NPP-1 sample		
Property	values	A	В	
Hydrazine diperchlorate, %	100.0	96.16	97.21	
Hydrazine monoperchlorate, %	0.0	2.41	0.65	
Hydrazine content, %	13.73	13.77	13.40	
Perchlorate content, %	85.41	83.97	_	
Total chloride, %	30.47	30.19	30.26	
Density, g/cm ³	! _ !	2.25	2.25	
Impact sensitivity, inlb.	_	68	68	





HP₂. Sample A was a pure white sample and Sample B was a discolored sample. Differential thermal analysis and spark sensitivity data will be reported in a subsequent issue of the SPS, Vol. V. Facilities for conducting these tests are presently being set up.

3. Chemical Analysis Methods¹

a. Total hydrazine content. Hydrazine is determined by the indirect iodate method. In the method employed here, an excess amount (50 ml) of a standard (0.1 normality) iodate solution is added to a known quantity (about 0.2 g by direct weighing or aliquot) of the sample in a total volume of 75 ml, and the iodine formed is boiled out. The reaction which takes place is described by the following equation:

$$5N_2H_4 + 4IO_3 + 4H \rightarrow 5N_2 + 2I_2 + 12H_2O$$
 (1)

In this case, no additional acid is necessary since hydrazinium diperchlorate already is sufficiently acid for the reaction to take place rapidly and quantitatively. In the course of experiments, it was observed that if the solutions were appreciably diluted, the first reaction proceeded more slowly and could conceivably lead to serious errors.

The excess iodate is then reacted with excess (1 to 2 g) of potassium iodide with 15 ml of 1:4 sulfuric acid, in accordance with:

$$IO_3^- + 5I^- + 6H^+ \rightarrow 3I_2 + 3H_2O$$
 (2)

The iodine liberated here is titrated with standard thiosulfate solution using a fresh starch solution as indicator.

$$I_2 + 2S_2O_3^* \rightarrow 2I^- + S_4O_6^*$$
 (3)

The thiosulfate solution was standardized against potassium dichromate using generated iodine; the iodate solution was standardized against the thiosulfate solution in a similar manner.

In these reactions the normal valence change for iodate is five; however, in the back-titration (Eqs. 2 and 3) all of the iodine does not come from the iodate, so the normality of the iodate solution here must be regarded as six times the molarity rather than five times. In the direct titration of hydrazine with iodate in the presence of strong hydrochloric acid, the normality of the iodate must be taken as four times the molarity. This change in the

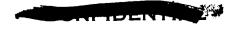
value of normality of the iodate can be regarded as exceptional in redox chemistry, and care should be taken to avoid using the wrong factor.

For a sample of pure hydrazinium diperchlorate, the hydrazine (N_2H_4) content is 13.73%; for pure hydrazinium monoperchlorate, 24.15%. Values between the two limits were obtained experimentally for different samples. Table 1 gives the total hydrazine content for two samples.

b. Total perchlorate content. A sample (0.2 g) of hydrazinium diperchlorate is placed (dry box operation) into a microfilter beaker, capped, and then weighed to the nearest 0.1 mg. Absolute ethanol is added (several milliliters) until the sample is completely dissolved, then a solution of alcoholic potassium hydroxide (made by dissolving KOH in absolute ethanol) is added until no more precipitate is formed. The supernatant liquid is then drawn off the precipitate by means of suction applied to the sintered glass filter, and the precipitate is washed several times with ethanol until a negative result is obtained in a test for excess potassium hydroxide; the assembly with the precipitate is then dried at 120°C to a constant weight. Since the entire operation (solution, precipitation, washing, and drying) is carried out in the special microfilter beaker, there is essentially no loss of precipitate in handling. For a pure sample of HP2 the theoretical perchlorate content is 85.41%. For pure hydrazine monoperchlorate the theoretical value would be 75.09%.

- c. Total chloride content. The total chloride content was determined gravimetrically by first fusing a sample (0.2 g) of hydrazinium diperchlorate with a large excess (2 g) of sodium carbonate in a platinum crucible, then dissolving the melt in water, acidifying with nitric acid, and precipitating silver chloride by the standard method.² For hydrazine diperchlorate and hydrazine monoperchlorate, the theoretical chloride content is 30.47 and 26.79%, respectively.
- d. Acid-hydrazine ratio and content. From hydrazine analyses, it is apparent that hydrazinium diperchlorate may contain hydrazinium monoperchlorate as an impurity. Inasmuch as the latter material is considerably more unstable with respect to shock sensitivity, etc., it is important to have a convenient, simple method of analysis (aside of total hydrazine content) which will indicate the relative amounts of the di- and mono-perchlorates in

²I. M. Kolthoff and E. B. Sandell, Textbook of Quantitative Inorganic Analysis, McMillan Co., New York, 1936.



¹The chemical analyses were conducted by I. Shapiro of Universal Chemical Systems Inc., Culver City, California.



a sample. For this purpose the acid-base titration curve is very useful.

Perchloric acid is a very strong acid; hydrazine is a weak base, serving primarily as a monoacid base. The dissociation constants of hydrazine at 25°C are reported³ as follows:

$$K_1 = \frac{\left[N_2 H_5^+\right] \left[O H^-\right]}{\left[N_2 H_4\right]} = 8.5 \times 10^{-7}$$
 (4)

$$K_2 = \frac{\left[N_2 H_6^{++}\right] \left[O H^{-}\right]}{\left[N_2 H_5^{+}\right]} = 8.9 \times 10^{-16}$$
 (5)

Because of the low value of the second ionization constant, diacid salts of hydrazine do not exist in aqueous solutions, but rather, the $N_2H_6^{++}$ ion reacts completely with the solvent water:

$$N_2H_6^{++} + H_2O \rightarrow N_2H_5^{+} + H_3O^{+}$$
 (6)

to yield free acid and the $N_2H_5^+$ ion. Consequently, the base titration curve of hydrazinium diperchlorate (dissolved in water) should show two breaks in the curve, corresponding to two endpoints, one representing the neutralization of H_3O^+ and the other representing the neutralization of $N_2H_5^+$:

$$N_2H_5^+ + OH^- \rightarrow N_2H_4 + H_2O$$
 (7)

The two-step curve is analogous to the titration of carbonate-bicarbonate mixtures, where the first endpoint represents the amount of free acid generated by the conversion of $N_2H_6^{++}$ to $N_2H_5^{+}$; and the second endpoint, by the conversion of $N_2H_5^{+}$ to N_2H_4 . For pure hydrazinium diperchlorate, the same volume of KOH required for the first endpoint will also be required to reach the second endpoint; any difference will then represent the amount of excess $N_2H_5^{+}$ over and above the amount of $N_2H_6^{++}$ originally present.

Using the ionization constant given in Eq. (4), the expected shape of the titration curve can be calculated, such as illustrated in Fig. 1. In this case, the normality (N_b) of the KOH was taken as 0.05 N; the initial weight of hydrazinium diperchlorate as 0.233 g in 100 ml (V_i) of solution $(C_{s_1} = 0.01 M)$. The equations used to calculate the various portions of the titration curve are shown in Fig. 1.

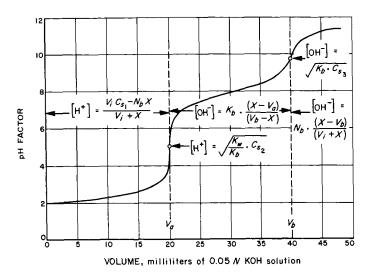


Fig. 1. Calculated pH-titration curve of 0.01 M hydrazinium diperchlorate ($V_i = 100 \text{ ml}$)

An inspection of the titration curve quickly reveals that the first endpoint is very distinct, but that the second endpoint is not distinct, and hence, can easily lead to errors in determining the true endpoint. The customary method of triangulation for determining the midpoint as the inflection point is subject to some uncertainty, depending upon the concentration of solutions used in the titration procedure. In the course of studying the acid-base titration, a more precise method for determining the second endpoint has been conceived, and is illustrated in Fig. 2. First, a known volume of perchloric acid solution is titrated with base (only one endpoint). Next, some

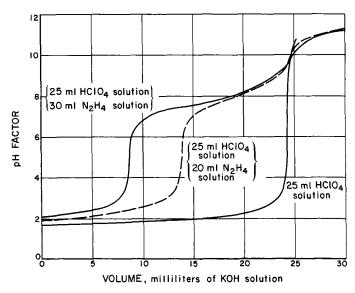


Fig. 2. Titration curve of perchloric acid with and without added hydrazine solution

³L. F. Andrieth and B. A. Ogg, *The Chemistry of Hydrazine*, John Wiley, New York, 1951.

hydrazine is added to an equal volume of perchloric acid solution, and again titrated with the same concentration of base, but in this case there will be two endpoints, with the second endpoint coinciding with the endpoint for perchloric acid alone. The first endpoint will be determined by the amount of hydrazine added. In the example given in Fig. 2, the differences between the first and second endpoints for the two samples containing hydrazine were in the exact ratio of the volumes of hydrazine added. The pH at the second endpoint can be determined precisely, and if concentrations and volumes of solutions are maintained within close limits, the pH can be used for determining experimentally the second endpoint. Thus, even if the pH instrument is not calibrated, it can be used effectively by this technique. The endpoint determined by this method does not necessarily coincide with the arbitrarily chosen midpoint of the curve.

The titration curves for Samples A and B are shown in Figs. 3 and 4. The weight of sample and the volumes at the two endpoints are tabulated alongside the titration curves. Using these data, the amount of hydrazinium diperchlorate (HP₂) and hydrazinium monoperchlorate (HP) present in the sample can be calculated. Additionally, the volume between the two endpoints is a function of the amount of the hydrazine in the sample. Caution should be exercised in the interpretation of the pH-titration data.

The pH-titration data yields information only on total hydrazine content and on total strong acid content, with the results relating back to the initial composition of the

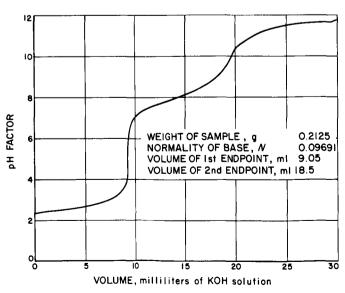


Fig. 3. pH-titration curve of Sample A (dry)

solid. However, it is possible for a sample of pure hydrazinium diperchlorate solid to absorb some moisture and hydrolyze, forming perchloric acid $(H_3O^+ \text{ClO}_4^-)$ and the monoperchlorate, and yet the pH-titration data would not reveal this condition. It is planned to supplement the titration data with infrared spectroscopy analysis. This technique should allow accurate determination of the final composition of the material.

e. Density. The solid density was measured with a benzene pycnometer at 30°C. To ensure proper degassing of the sample in the liquid, a vacuum was formed in the pycnometer when it was half-filled with the benzene, thus removing adsorbed gases. The density was found to be 2.25 g/cm³.

f. Impact sensitivity. A rather extensive study has been made of the sensitivity to impact of the various lots of HP₂ currently under study. The effect of moisture and residual solvent on the impact sensitivity has also been studied. As previously mentioned, the material from Lots NPP-1 and NPP-2 is stored under CCl₄. In preparation for use, the HP₂ sample containers are opened in a dry box, the CCl₄ filtered off, and the HP₂ powder vacuum-dried to remove any residual solvent. All the sample preparations for the impact tests are conducted in a dry box and transferred to the test apparatus in a desiccator. Tests have been run on samples subjected to moist atmosphere, on samples containing residual solvent, and on samples of agglomerated and caked material

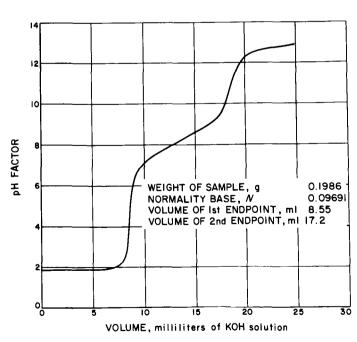


Fig. 4. pH-titration curve of Sample B





Table 2. Impact sensitivity of HP2, AP, and JPL-450

Sample	Sensitivity drop height, in.	
AP, unground ^a	33.4	
AP, ground*	26.7	
AP, spherical ^c	28.1	
PL-540 propellant	24.0	
fP2, Lot NPP-1, dry white	17±1.5	
IP2, Lot NPP-1, dry discolored	1 <i>7</i> ±1.5	
IP2, Lot NPP-1, agglomerated	14生0.5	
IP₂, Lot NPP-1, residual CCl₄	1 <i>7</i> ±1.5	
IP2, Lot NPP-2, dry	19±1.0	
IP₂, Lot NPP-2, residual CCI₄	1 <i>7</i> ±0.5	
IP ₂ , Lot NPP-2, agglomerated	13.5±1.5	
IP ₂ , Lot TCC-1, dry	16.5 <u></u> ±0.5	
tP₂, Lot TCC-1, moist ^d	>36	
fP ₂ , Lot NPP-2, moist ^e	28	
IP ₂ , Lot NPP-2, moist ^f	>48	

^a Average particle size \approx 170 μ .

screened out of the dry HP₂ powder. All impact tests were run on the JPL tester using the Bruceton (up-and-down) method. The drop weight was a 4-lb ball. Table 2 shows the impact sensitivity data for the various samples of HP₂, as well as impact sensitivity of ammonium per-chlorate and JPL-540 propellant (included here as reference data). All data is reported as the estimated mean of 50% point. Each mean value is determined on the basis of 16 to 50 specimens per sample.

As will be noted in Table 2, essentially no difference in impact sensitivity occurs between the dry white, dry discolored, or CCl₄ wet material. However, the agglomerated material did appear somewhat more sensitive. One possible explanation is that some decomposition has occurred within the sample, and the decomposition products are causing the agglomerations to occur. Any decomposition products would contain an increased monoperchlorate content. The range in drop height for the 50% point is due to the variation in values obtained from samples out of different containers within the same lot of HP₂.

4. Moisture Effects

No quantitative data on the effects of moisture on the properties of the HP₂ are reported here. However, pre-

liminary tests indicate a greater tendency of the HP_2 salt to form the dihydrate than to decompose to the monoperchlorate and free perchloric acid. Samples subjected to various relative humidities (RH) appear to pick up sufficient moisture to form the dihydrate very rapidly, at which time the rate of moisture pickup becomes rather slow. Above RH=45% the salt becomes liquid in a few minutes, whereas below RH=35% the material remains as a powder for several hours.

B. Applications Technology Satellite Motor Development

R. L. Haserot

1. Introduction

In January 1963 JPL initiated a development program to provide a solid propellant apogee rocket motor for a second-generation *Syncom* satellite. This program, under the management of the Goddard Space Flight Center, was designated Advanced *Syncom*. It was to produce a spin-stabilized, active repeater communications satellite, weighing about 750 lb, and operating at synchronous altitude (22,300 mi); the satellite would handle voice communications, teletype, and monochrome and color television signals.

In January 1964 the Advanced Syncom communication program was redirected to include a number of experimental instruments in addition to the original communication instruments. This expanded program is the Applications Technology Satellite (ATS)4 Program and will produce a general purpose satellite capable of operating at synchronous altitude with experimental instruments in the areas of meteorology, communications, radiation, navigation, gravity-gradient stabilization, and engineering technology. For those satellites to be placed in synchronous orbit, JPL will provide a solid propellant rocket motor to provide the final required velocity increment at the apogee of the elliptical transfer orbit. This rocket motor is designated the JPL SR-28-1 (steel chamber) or IPL SR-28-3 (titanium chamber) rocket motor. It is presently intended that only the JPL SR-28-3 unit will be delivered for flight use.

Previous reports of progress on the development of this motor have been published in SPS 37-20 to 37-30, Vol. V.

 $^{^{}b}$ Average particle size \approx 17 $\mu.$

 $[^]c$ Average particle size pprox 180 μ .

 $[^]d$ Samples conditioned for 3 hr at RH = 35%.

^{*}Samples conditioned for 1 hr at RH = 35%.

 $^{^{}f}$ Samples conditioned for 3 hr at RH = 35%.

⁴ Formerly designated Advanced Technological Satellite (ATS).

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2. Program Status

The motor development program calls for static firing of four heavywall motors and 20 flightweight motors, including three with flight-design titanium chambers, prior to conducting a nine-motor qualification program. To date the four heavywall motors plus 12 flightweight motors have been static fired, two of which were fired under simulated high-altitude conditions at Arnold Engineering Development Center (AEDC) Tullahoma, Tennessee. All of the flightweight motors tested to date have been with Type 410 chromium steel chambers. Detailed descriptions and test data on a spin-firing and two temperature tests are included in this article.

3. Flightweight Motor Static Firings

a. Development Test G-5. Motor P-16 was static fired November 24, 1964 to evaluate the effects of spinning on motor hardware and internal ballistics. The motor assembly consisted of flight-design components throughout except that the nozzle exit cone had an expansion ratio of 8.5. The motor was conditioned to a temperature of 60°F prior to installation on the spin stand. The motor was fired while spinning at 150 rpm about the thrust axis. Instrumentation included two channels of chamber pressure and one channel of igniter pressure (taken through slip rings) and a spin-rate counter.

The motor ignited and burned normally for full duration. There was no appreciable change in the spin rate during motor burning. A summary of motor internal ballistic data is shown in Table 3. By comparing these results with results of motors tested at the same temperature, it is apparent that internal ballistic performance was unaffected by the spin rate of 150 rpm.

Examination of motor hardware was made after the test and compared to that of motors fired while not spinning. A comparison of motor hardware conditions is shown in Table 4. The chamber insulation was in slightly better condition due to uniform heating caused by the spinning. The ignition system lost slightly more weightprobably due to centrifugal force throwing the aluminum oxide deposits into the gas stream. Significant differences were observed in the nozzle. The general condition of the nozzle from the spin test was much better than for nonspinning tests. Although the weight lost during test was unchanged, throat erosion was slightly less, erosion of the entrance section was minor, throat concentricity was better, and the entire nozzle was relatively free from aluminum oxide deposits. Figs. 5 and 6 show the postfiring condition of nozzles which were tested while spinning and not spinning, respectively.

b. Development Test G-1. Motor assembly P-11 was static fired December 16, 1964 after being subjected to

Table 3. ATS apogee motor static test summary

Test code designation	G-1	G-2	G-5
Motor No.	P-11	P-10	P-16
Run No.	E-342	E-343	E-326
Conditioning temperature, °F	110	10	60
Primary test objectives	Temperature cycling	Temperature cycling	Evaluate performance while spinning at 150 rpm
Chamber configuration	Flight; steel	Flight; steel	Flight; steel
Insulation configuration; weight, 1b	Flight; 10.6	Flight; 10.4	Flight; 10.4
Nozzle expansion ratio; weight, lb	$\epsilon=$ 35; 37.2	ε = 35; 37.6	ϵ $=$ 8.3
Propellant weight, Ib	766.8	766.1	768.7
Run time, sec	41.3	45.5	43.1
Initial chamber pressure, psia	100.9	98.0	98.9
Maximum chamber pressure, psia	277.1	242.7	258.9
Nozzle throat area increase, %	1.54	1.55	1.45
W* (avg) performance, ft/sec	4976	4949	4966



•

Table 4. Comparison of postfiring motor hardware conditions for spinning and nonspinning static tests

Hardware item and parameter	Spinning (Test Code G-5)	Nonspinning (average of six tests)	Hardware item and parameter	Spinning (Test Code G-5)	Nonspinning (average of six tests)
Chamber insulation: weight lost during testing, lb	1.21	1.87	Throat concen- tricity, in.	0.0035	0.0072
Ignition system: weight lost during testing, lb Nozzle (expansion ratio = 8.5)	1.09	1.04	Appearance of graphite entrance nose	Smooth; very little erosion	Minor localized erosion and rutting; nose eroded back approximately 0.05 in.
Weight lost during testing, lb	1.73	1.74	Appearance of exit	Relatively free of	Significant amounts
Throat area increase, %	1.45	1.72	cone and outside of buried portion	aluminum oxide deposits	of aluminum oxide deposits



Fig. 5. Nozzle postfiring condition; tested while spinning

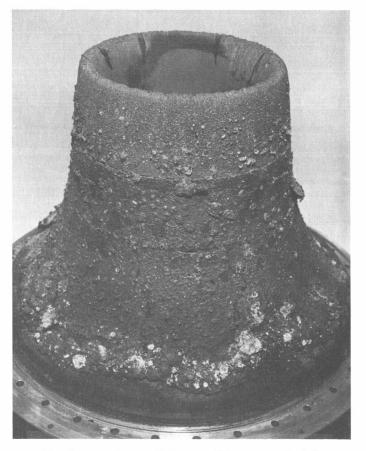


Fig. 6. Nozzle postfiring condition; tested while not spinning



temperature cycling and conditioning according to the schedule in Table 5. Prior to final temperature cycling at 110°F, the motor received thorough visual and radiographic inspection. This motor was cast from three batches of propellants mixed in the 25-gal Bramley mixer. All motor components were of flight design.

The motor was fired into the atmosphere while mounted on the horizontal test stand at the JPL-ETS facility. The motor ignited and burned normally for full duration. Motor performance data is summarized in Table 3.

Table 5. Schedule of temperature cycling for Development Tests G-1 and G-2

Test code designation	G-1	G-2
Temperature cycles, °F	110	10
1	10	110
	110	10
Visual and radiographic inspection	~	"
Shipping temperature cycle ^b , °F	10	110
	110	10
	10	110
	110	10
	10	110
	110	10
	10	110
	110	10
	10	110
ł	110	10
Visual and radiographic inspection	~	"
Final temperature conditioning (72 hr), °F	110	10

⁵24-hr duration at each temperature

c. Development Test G-2. Motor assembly P-10 was static fired December 16, 1964 after being subjected to temperature cycling and conditioning according to the schedule on Table 5. Prior to final temperature conditioning at 10°F, the motor received thorough visual and radiographic inspection. Inspections during the temperature cycling and the final inspection revealed areas of insulation/propellant unbonding and separation around the igniter and nozzle openings of the chamber. At the end of temperature cycling, the separation at the igniter opening was ¼ in., the approximate amount which would be caused by propellant grain shrinkage on curing. The motor had been cast from a single batch of propellant mixed in the 150-gal Baker-Perkins mixer. A review of the physical properties of the propellant cast into the motor showed higher than usual tensile strength and modulus. However, this alone would not normally be severe enough to cause a separation of properly bonded propellant. A possible alternate cause of the unbonding would be contamination of the insulation surface prior to casting.

To prevent a recurrence of separations, the processing procedures were revised to reduce the chance of insulation-contamination. In addition the propellant-binder cross-link ratio has been adjusted to give a propellant with lower tensile strength and modulus.

Motor P-10 was repaired by filling the separation with epoxy adhesive; subsequent X-ray of the repaired area showed no separations. The motor was mounted on the horizontal test stand at the JPL-ETS facility and fired into the atmosphere, igniting and burning normally for full duration. Motor performance is summarized in Table 3.